



NRL/MR/6355--15-9633

Sensitization of Naturally Aged Aluminum 5083 Armor Plate

RONALD L. HOLTZ
RAMASIS GOSWAMI
PETER S. PAO

*Multifunctional Materials Branch
Materials Science and Technology Division*

July 29, 2015

Approved for public release; distribution is unlimited.

REPORT DOCUMENTATION PAGE				Form Approved OMB No. 0704-0188	
Public reporting burden for this collection of information is estimated to average 1 hour per response, including the time for reviewing instructions, searching existing data sources, gathering and maintaining the data needed, and completing and reviewing this collection of information. Send comments regarding this burden estimate or any other aspect of this collection of information, including suggestions for reducing this burden to Department of Defense, Washington Headquarters Services, Directorate for Information Operations and Reports (0704-0188), 1215 Jefferson Davis Highway, Suite 1204, Arlington, VA 22202-4302. Respondents should be aware that notwithstanding any other provision of law, no person shall be subject to any penalty for failing to comply with a collection of information if it does not display a currently valid OMB control number. PLEASE DO NOT RETURN YOUR FORM TO THE ABOVE ADDRESS.					
1. REPORT DATE (DD-MM-YYYY) 29-07-2015		2. REPORT TYPE Memorandum Report		3. DATES COVERED (From - To) December 2010 – December 2014	
4. TITLE AND SUBTITLE Sensitization of Naturally Aged Aluminum 5083 Armor Plate				5a. CONTRACT NUMBER	
				5b. GRANT NUMBER	
				5c. PROGRAM ELEMENT NUMBER 61153N	
6. AUTHOR(S) Ronald L. Holtz, Ramasis Goswami, and Peter S. Pao*				5d. PROJECT NUMBER	
				5e. TASK NUMBER	
				5f. WORK UNIT NUMBER 4297	
7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) Naval Research Laboratory, Code 6350 4555 Overlook Avenue, SW Washington, DC 20375-5328				8. PERFORMING ORGANIZATION REPORT NUMBER NRL/MR/6355--15-9633	
9. SPONSORING / MONITORING AGENCY NAME(S) AND ADDRESS(ES) Office of Naval Research One Liberty Center 875 North Randolph Street, Suite 1425 Arlington, VA 22203-1995				10. SPONSOR / MONITOR'S ACRONYM(S) ONR/NRL	
				11. SPONSOR / MONITOR'S REPORT NUMBER(S)	
12. DISTRIBUTION / AVAILABILITY STATEMENT Approved for public release; distribution is unlimited.					
13. SUPPLEMENTARY NOTES *Formerly at NRL					
14. ABSTRACT A sample of aluminum armor plate, approximately 40 to 50 years old, containing a large intergranular stress corrosion crack was evaluated. Scanning electron microscopy examination of the fracture surfaces is consistent with intergranular stress corrosion cracking and some evidence of variable environmental and stress history is noted. ASTM G-67 nitric acid mass-loss values were 19 to 25 mg/cm ² . The transmission electron microscopy microstructure of the sample was found to be consistent with sensitization due to aging at low temperatures for a long time. The grain boundary magnesium-rich phase is continuous, although non-uniform, and only 10 to 15 nm thick.					
15. SUBJECT TERMS Sensitization Aluminum armor Aluminum 5083					
16. SECURITY CLASSIFICATION OF:			17. LIMITATION OF ABSTRACT Unclassified Unlimited	18. NUMBER OF PAGES 9	19a. NAME OF RESPONSIBLE PERSON Ronald L. Holtz
a. REPORT Unclassified Unlimited	b. ABSTRACT Unclassified Unlimited	c. THIS PAGE Unclassified Unlimited			19b. TELEPHONE NUMBER (include area code) (202) 767-6231

CONTENTS

ABSTRACT	iv
INTRODUCTION	1
SAMPLE DESCRIPTION	1
SEM EXAMINATION	2
ASTM G-67 MASS LOSS TEST	2
TEM MICROSTRUCTURE	3
CONCLUSION	4
ACKNOWLEDGEMENTS	5
REFERENCES	6

FIGURES

Figure 1. Armor plate and sample locations	1
Figure 2. SEM micrographs of crack faces	2
Figure 3. TEM of typical grain boundaries	3
Figure 4. Fine probe EDS line scan across grain boundary	4
Figure 5. Composition maps of intragranular particles	4
Figure 6. Mg rich phases in the bottom half of the plate	5

SENSITIZATION OF NATURALLY AGED ALUMINUM 5083 ARMOR PLATE

INTRODUCTION

Aluminum-magnesium alloys are important for both ship structures [1], and land vehicle armor [2]. These 5xxx alloys Al-Mg solid solution alloys, containing three to six percent magnesium, along with some manganese, exhibit high strength, excellent corrosion resistance, and are weldable. However, under prolonged exposure to temperatures of 50 to 200 °C, these alloys can degrade by the mechanism of sensitization, which occurs when magnesium normally in solid solution in the alloy diffuses to the grain boundaries [3,4]. The magnesium-rich phase (normally β -Al₃Mg₂) is highly anodic with respect to the surrounding aluminum phase, thus is susceptible to intergranular corrosion.

While there have been several studies of sensitization under controlled, artificial aging, usually at elevated temperatures up to 175 °C, little detailed information exists in the literature on very long-term aging at low or ambient operational temperatures. Recently, we had the opportunity to examine a piece of aluminum 5083 armor plate taken from an aging armored vehicle. The vehicle from which it was removed had been in normal service for 40 to 50 years. Unfortunately the exact environmental and thermal history of this plate are unknown, but likely there was environmental exposure to humid air, rain, saltwater, and engine exhaust; and elevated temperatures due to solar loading and proximity to the vehicle's engine. The plate contained a large delamination crack in the mid-plane of the plate. The objective of this study was to identify if the crack in this sample was associated with sensitization.

SAMPLE DESCRIPTION

The 5083 plate sample is shown in Figure 1. The temper designation was not known, but armor plate often is H131 temper. Several specimens for transmission electron microscopy (TEM) were cored out of the region ahead of the crack tip. The specimens for the ASTM G-67 mass loss test were taken from the end of the sample as indicated. The front of the sample was cut off about one inch behind the crack tip, and the crack faces examined by SEM.

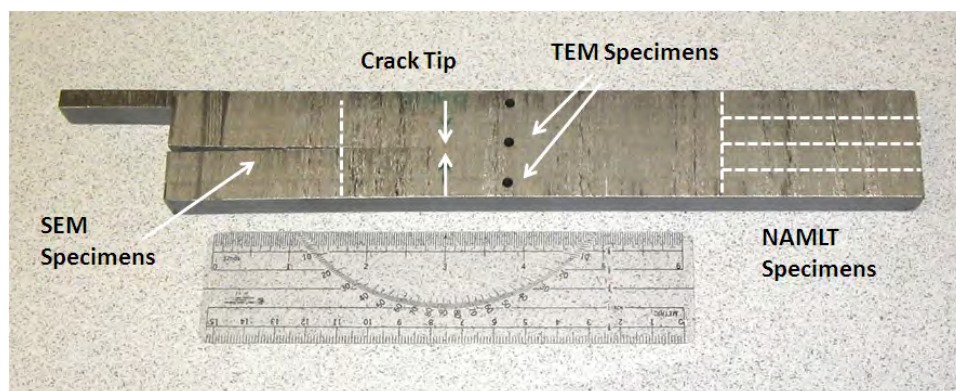


Figure 1. Armor plate and specimen locations.

SEM EXAMINATION

A cursory examination of randomly selected regions of crack faces was done with SEM. Most regions of the crack face, such as Figure 2a, exhibit clear, well defined flattened grain surface typical of intergranular crack growth of rolled aluminum alloys, and with varying amounts of debris scattered about the surface consistent with corrosion product, Figure 2b, that often forms over time within a crack. Figure 2c shows a region with a very thick layer of corrosion product in a “mud-crack” pattern, and may represent exposure to a particularly aggressive corrosive attack at some point in the sample’s history. There are other regions around the crack face, Figure 2d, that are consistent with a ductile fracture mechanism, perhaps as the result of some overload event.

On the basis of SEM examination, generally our assessment is that the crack appearance is consistent with intergranular stress corrosion cracking (IGSCC), although corrosion-fatigue cannot be ruled out on the basis of the SEM examination alone. Variations in the corrosion product and fracture mode would be consistent with a part that had seen a range of environment-stress conditions throughout its lifecycle.

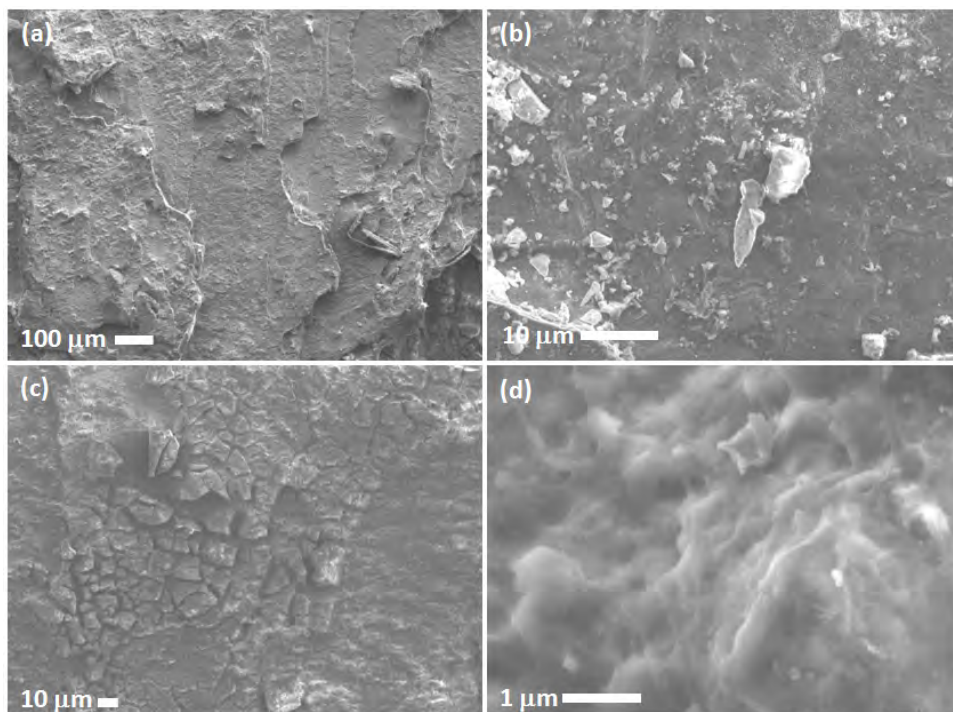


Figure 2. SEM micrographs of the crack faces showing (a) typical flattened grain intergranular cracking mode, (b) corrosion debris on the crack surface, (c) mud-crack pattern indicating aggressive environment exposure, and (d) localized region of ductile fracture.

ASTM G-67 MASS LOSS TEST

The ASTM G-67 “Standard Test Method for Determining the Susceptibility to Intergranular Corrosion of 5XXX Series Aluminum Alloys by Mass Loss after Exposure to Nitric Acid” was used as an assessment of the degree of sensitization (DOS) of the alloy.[5] Three ASTM G-67 tests were done. The mass-loss results were 19, 20 and 25 mg/cm² with an average value of 21.3 mg/cm². The highest of the three values was a specimen from the bottom surface of the sample (the side facing the engine). The other two values were

from the middle of the sample. One other specimen from the top surface of the sample also was measured, yielding a mass loss of 27 mg/cm²; however, the test conditions on this specimen deviated from the standard test method so this result should not be regarded as valid.

TEM MICROSTRUCTURE

Sample preparation and the TEM and HRTEM techniques used are described in our several publications. [6,7] We examined two specimens cored from approximately 25 mm in front of the crack tip. One specimen was in the mid-plane of the sample; i.e., in the same plane as the crack, and the other was from the bottom of the sample; i.e., the side closest to the engine. A specimen from the top portion of the plate was not examined.

Figure 3 shows TEM images of typical grain boundaries in the mid-plane specimen. Figure 3(a) is a bright-field image showing that a continuous grain boundary phase with a thickness of about 10 nm is present. Figure 3(b) is a Z-contrast image, which displays the lighter elements darker. This shows that the grain boundary phase is composed of lighter elements than the aluminum matrix. Since the only constituent in the alloy lighter than aluminum is magnesium, it is reasonable to conclude that the grain boundary phase is magnesium rich. The fine-probe energy dispersive spectrometry (FPEDS) line scan in Figure 4 confirms the grain boundary is magnesium rich, over an extent of 10 to 20 nm, and the aluminum concentration in this region is lower. The average concentration indicated of about 19% Mg is somewhat lower than β , which would be 40%, but this is most likely an averaging artifact since the thickness of the grain boundary phase is very small and the Mg phase may not be perfectly edge-on.

We did not specifically confirm this magnesium rich phase to be β -Al₃Mg₂. The thickness is too small to use diffraction in the conventional TEM, thus would require use of high-resolution TEM and lattice plane imaging. However, we are confident that this is in fact β phase because: (1) β is the only equilibrium Al-Mg phase known to exist at these Mg concentrations; and (2) all of our other work on 5083 alloy identifies β phase as the magnesium rich grain boundary phase even when the precipitates are very small.

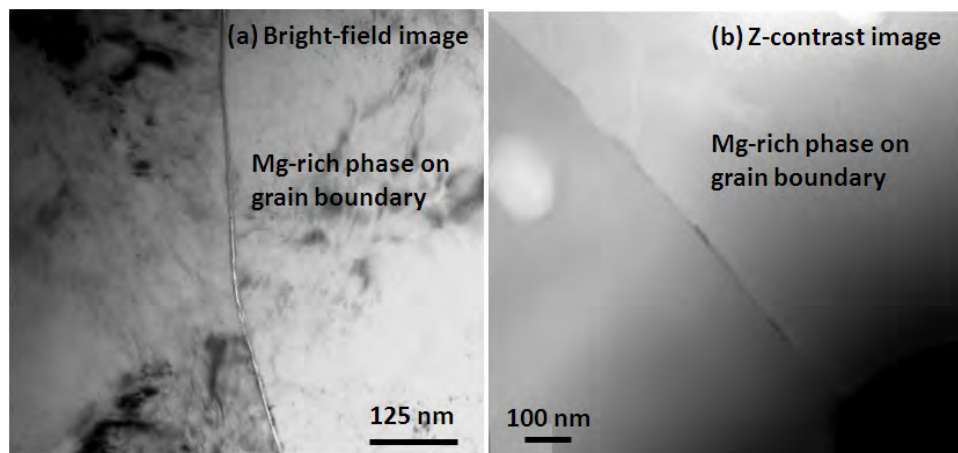


Figure 3. TEM images of typical grain boundary and grain boundary phase in the mid-plane specimen: (a) bright-field image and (b) Z-contrast image. In (b), darker areas indicate lighter elements and vice-versa.

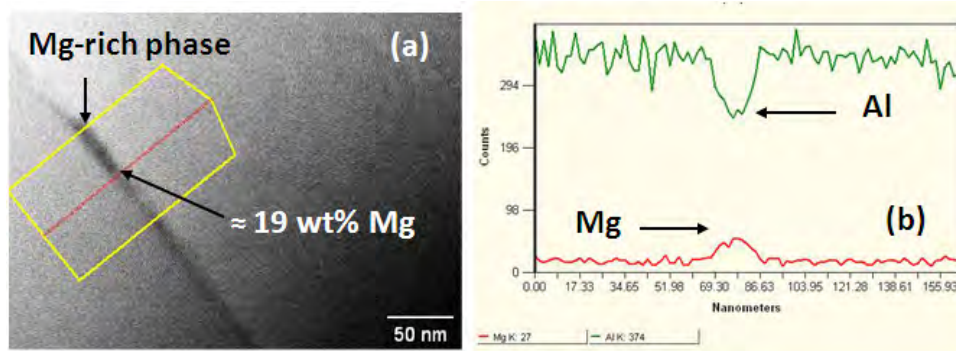


Figure 4. Z-contrast image (a) and corresponding fine-probe EDS line scan (b), showing that the grain boundary phase is Mg-rich.

In addition to Mg-rich phase forming on the grain boundaries, we also see Mg-rich phase precipitating in very thin layers (less than 10 nm) on rod-shaped second-phase particles (typically Al_6Mn structure but also containing Fe or Cr) present in the interiors of grains. See Figure 5. We have seen this commonly in our other work on 5083 alloy.[3] Mg present on intragranular particles is not expected to contribute to IGSCC, but may promote pitting damage.

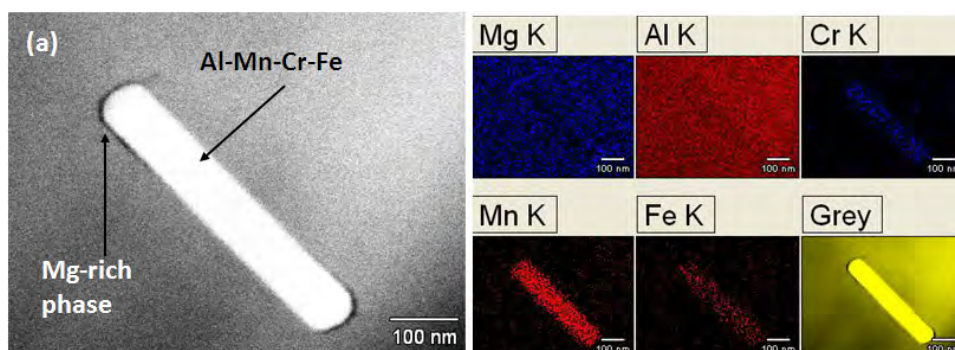


Figure 5. Z-contrast image (a) and corresponding fine-probe EDS composition maps showing of an intragranular particle with a thin precipitated layer of Mg.

The specimen from the bottom half of the plate also had Mg-rich (probably β) on the grain boundaries, but was generally not continuous. Figure 6(a) and 6(b) shows the Z-contrast image and FPEDS line scan indicating the Mg-rich grain boundary phase. The higher resolution images of Figure 6(c) and 6(d) show a discontinuous grain boundary structure, very similar to what we have observed in other studies to be characteristic of the evolution of β phase prior to it forming continuous coverage.[4] Figure 6(d) shows Mg-rich phase (probably β) on an intragranular particle, just as occurs in the mid-plane region of the sample.

CONCLUSION

The microstructure of the naturally aged, cracked 5083 armor plate sample is consistent with sensitization due to aging at low temperatures for a long time. The grain boundary phase in the mid-plane of the plate is continuous, but non-uniform, and very thin, only 10 to 15 nm thick. ASTM G-67 tests showing mass loss results of around 19 - 25 mg/cm² are consistent with at least an intermediate degree of sensitization, but somewhat lower than expected for continuous β coverage of the grain boundaries.

Generally, the results suggest that even an extremely thin layer of β below the resolution of conventional optical and SEM examination, and a mass loss that is not normally considered risky, can be associated with significant cracking

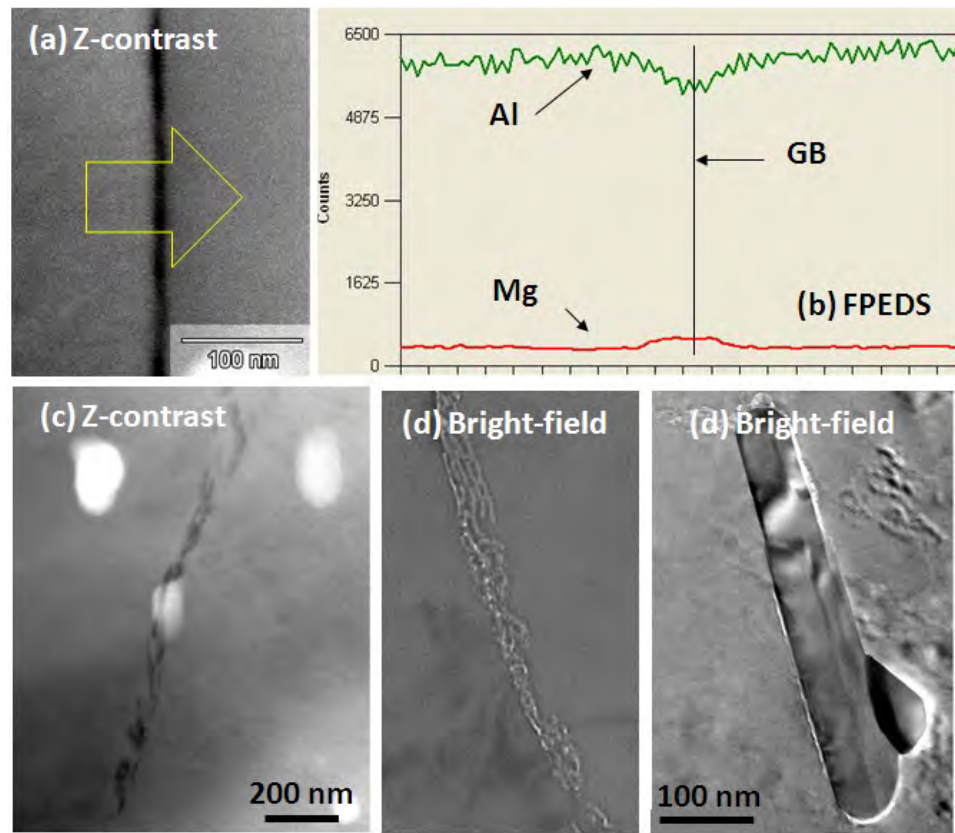


Figure 6. Mg-rich phase precipitation in the specimen in the bottom half of the plate. (a) Z-contrast image and corresponding fine-probe EDS line scan (b). Images (c) and (d) showing the discontinuous morphology and (d) a bright-field image indicating Mg precipitation on an intragranular particle.

ACKNOWLEDGEMENTS

Dr. Robert Bayles, formerly of the Center for Corrosion Science and Engineering of the Naval Research Laboratory, assisted in the planning of these tests. Mr. Thomas Longazel, formerly of the Center for Corrosion Science and Engineering of the Naval Research Laboratory, performed the ASTM G67 testing. The authors are grateful to Mr. Leroy Levenberry, Leidos, Inc., for technical support on this project. Funding for this project was provided by the Office of Naval Research (ONR) through the Naval Research Laboratory's Basic Research Program

REFERENCES

- [1] “Protecting the future forces”, J.S. Montgomery and E.S. Chin, The AMPTIAC Quarterly, 8, #4, 15-20, (2004).
- [2] “Research needs in aluminum structures”, R.A. Sielski, Ships and Offshore Structures, 3, 57-65 (2008).
- [3] “Stress corrosion cracking of sensitized AA 5083, J.L. Searles, P.I. Gouma, and R.G. Buchheit, Metallurgical and Materials Transactions A, 32A, 2859-2867 (2001).
- [4] “Sensitization and environmental cracking of 5xxx aluminum marine sheet and plate alloys”, F. Bovard, in Corrosion in Marine and Saltwater Environments II, edited DA Shifler, T. Tsuru, PM Natishan, S Ito, Electrochemical Society Proceedings, Volume 2004-14, pp. 232-243, Electrochemical Society (2005).
- [5] “Standard test method for determining the susceptibility to intergranular corrosion of 5XXX series aluminum alloys by mass loss after exposure to nitric acid (NAMLT Test)”, ASTM G-67-04.
- [6] “Precipitation behavior of the β phase in Al-5083”, R. Goswami, G. Spanos, P.S. Pao and R.L. Holtz Materials Science and Engineering A, 527, 1089-1095 (2010).
- [7] “Microstructure evolution and stress corrosion cracking behavior of Al-5083”, R. Goswami, G. Spanos, P.S. Pao, and R.L. Holtz, Metallurgical and Materials Transactions A, 42A, 348-355 (2011).
- [8] “Transmission Electron Microscopic Investigations of Grain Boundary Beta Phase Precipitation in Al 5083 Aged at 373K”, R. Goswami and R.L. Holtz, Metallurgical and Materials Transactions A, 44A, pp. 1279-1289 (2013).
- [9] “Corrosion Fatigue of Al 5083-H131 Sensitized at 70, 100 and 175 °C and Relation to Microstructure and Degree of Sensitization”, R.L. Holtz, P.S. Pao, R.A. Bayles, T.M. Longazel, and R. Goswami, Proceedings of the DoD Corrosion Conference, La Quinta, California, August 2011, NACE International, (2011).